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Large Scale Fixed Bed Ion-Exchange System for Removing Strontium-90 from Fluid Milk. I. Processing Results'

E. M. SPARLING, E. J. BALDI, R. O. MARSHALL, and B. HEINEMANN Producers Creamery Company, Springfield, Missouri

H. E. WALTER

Dairy Products Laboratory, Eastern Utilization Research and Development Division, USDA Washington, D. C.

and

JACK H. FOOKS

Research Branch, Division of Radiological Health, Public Health Service, U. S. Department of Health, Education, and Welfare Washington, D. C.

Abstract

A large scale fixed bed ion-exchange system for removing Sr[®] from fluid milk is described. The plant was operated over a period of ten months, processing a total of 413,000 liters of milk at flow rates of 5,700 liters/hour during an 8-hr run. No unusual processing problems were encountered.

The plant was fully automated (except for re-use of regenerant), constructed of stainless steel and other approved materials, and in compliance with 3-A Standards where applicable. Processing conditions were based on data obtained at Beltsville for flow rates, pH adjustment, regenerant composition, and sanitation control. The processed milk, although organoleptically acceptable, was spraydried and utilized for animal feed. In the event that environmental contamination should exceed safe tolerances, this process is a feasible and practical means for substantially reducing the levels of Sr⁵⁰ in milk.

The Beltsville pilot plant described by Edmondson et al. (1) and the process followed there were scaled up to be capable of processing 45,400 liters of milk per 8 hr, or 90,000 liters of milk per 16-hr day (2). The plant was fully automated, except for the re-use of regenerant. A succeeding report (3) describes changes in composition as a result of processing milk for the removal of Sr. A description of the commercial plant and its operation are discussed in this report.

Materials and Methods

Plant description. A commercial plant was designed using 3-A Standards and following the U. S. Public Health Standard Ordinance and Code wherever applicable. The plant has a rated capacity of 5,700 liters/hour of milk for an 8-hr run. Two 22,000-liter storage tanks were utilized, one for raw milk prior to processing, and one to receive pasteurized milk after removal of Sr⁸⁰.

The raw milk was pumped with a positive pump from the storage tank at the rated capacity, through a plate heat exchanger to bring it to about 13 C. The citric acid solution (ca. 20%) was pumped with a variable-drive positive pump (ca. 57 1/hr) through plastic tubing into the eye of a centrifugal line mixer, to achieve rapid and complete mixing of milk and acid solution. The volume of acid injected was regulated by a pH controller, to maintain the pH of the milk at 5.35. The acidified milk then passed through one of two in-line filters to one of two stainless steel columns. The filters were alternated if required, because of increasing pressure, by means of pressure activated valves. These columns were custommade, 107 cm in diameter by 244 cm high (488 cm with legs), and contained 1,325 liters IR-120 resin.

After passing downward through the resin bed, the milk was neutralized to pH 6.8, using 6% potassium hydroxide solution fed by a variable-drive positive pump (210 1/hour) into the eye of a centrifugal pump. After pH adjustment, the milk was allowed to accumulate in a surge tank for about 15 min prior to pumping with a positive pump through a plate heat exchanger (using regeneration) to pasteurize the milk. It was then cooled and stored cold until it was spray-dried. Although the processed milk was satisfactory organoleptically, it was all diverted to animal food use.

The capacities, in liters, of the polyethylenelined milk steel tanks were 9,500 for the regenerant, 800 for the citric acid, and 2,000 for the potassium hydroxide solution. The entire system was fully automated except for regeneration sequencing, which was manually

¹A report of work done under contract with the U. S. Department of Agriculture and authorized by the Research and Marketing Act of 1946. The contract was supervised by the Eastern Utilization Research and Development Division of the Agricultural Research Service. Funds were jointly provided by Agricultural Research Service and the Division of Radiological Health of the Public Health Service, USDHEW.

operated to reduce the number of regenerant tanks required. The processing equipment was installed in an area 7.3 by 18.3 m, in a room with a ceiling height of 4 m.

Plant procedures. Fresh Grade A whole milk standardized to 3.6% butterfat or skimmilk was delivered to the plant for each run. The plant was started on 2 C water, the resin bed cooled to 13 C, and the preheater regulated to maintain the milk temperature at 13 C. Milk was then circulated through the pH electrode system and the preheater back into the raw milk storage tank until both the temperature and the pH controlling system became stabilized. The pH of the milk was verified by laboratory measurements. Milk was then diverted to forward flow through the column, the neutralizing system, the surge, the pasteurizing system, and finally into a storage tank.

At the completion of the milk run, water was introduced into the system to rinse the resin. After a preliminary rinse, the flow was reversed and a nonionic detergent added to aid in removing milk solids from the resin. Temperature of the water was increased until the column effluent reached 48 C. After the detergent wash was completed, the column was rinsed at 48 C and finally brought to a temperature above 82 C.

Not more than 24 hr before the next run, the column was rinsed with water before regeneration was initiated.

Based upon preliminary bench scale trials, e composition of the regenerant solution described by Edmondson et al. (1) was modified slightly, as shown in Table 1.

Table 1

Chemical	Kg/BV of regenerant
Calcium chloride (anhydrous) Potassium chloride Sodium chloride Magnesium chloride hexahydrate Hydrochloric Acid	52.20 27.17 12.75 24.54 0.64

The last portion [4.5 or 9 BV (Bed Volume²)] of regenerant solution used on the previous run was passed through the column at 60 C at a flow rate of about 75 liters/min. This portion of the regenerant was directed to waste. The freshly prepared portion (4.5 or 9 BV) was heated to 60 C, passed through the column, and stored for the succeeding run. The column was then rinsed with water until all traces of regenerant salts were removed and the column chilled to 13 C with 2 C water immediately before using.

Results and Discussion

A total of 144,000 liters of Grade A skimmilk and 269,000 liters of Grade A whole milk were

processed during the period of May 22, 1964, to February 25, 1965. (A preliminary run of 10,000 liters of skimmilk was made May 21, 1964, to test the operation of the equipment. No compositional data were obtained on this run.) As discussed in the succeeding report, Sr® removal averaged 91.7% for these runs. No major processing problems were encountered, thus indicating the soundness of the pilot plant work done at Beltsville (1).

Flow rates of 45,400 liters of milk per 8-hr run were attained without difficulty and flow rates perhaps could be increased to reduce processing time to 6 hr, using the same equipment.

During the first runs, there was a solids buildup on the plates of the HTST pasteurizer which made it necessary to reduce milk flow rates to the pasteurizer near the end of the run. This problem was overcome by using a larger surge tank between the neutralizing line mixer and the pasteurizer. This permitted a greater uniformity in the pH of the milk going to the pasteurizer, which reduced the solids build-up to a level normally expected in a milk pasteurizer.

In this plant, the citric acid, potassium hydroxide, and regenerant solution tanks were not equipped for mechanical agitation. Experience showed the importance of maintaining homogeneity in these chemical solutions. Future plants, therefore, should provide for mechanical agitators in these vessels. Experience also dictates use of a reliable bench-model pH instrument for standardizing and checking the in-line pH electrodes at frequent intervals. It was also found necessary to add hydrochloric acid, to bring the pH of the regenerant solution to 5.6, to eliminate interaction of the regenerant salts and precipitation in these solutions

Instrumentation for the system was designed to give a semiautomated system. The engineering and functional aspects performed well. It was felt, however, that unless the system is completely automated, mechanical sequencing should be replaced by manual sequencing. In this case, the operator establishes the processing conditions as needed. There must be one operator present during operation of the equipment. He can completely monitor the plant, making such pH check tests, solution additions, and other routine observations and adjustments without being overly burdened. It is felt that unit process operators in a modern dairy processing plant will experience no difficulty in learning to operate this ion-exchange equipment.

Since regenerant costs represent a substantial portion of the total costs of the process, efforts were made to reduce the amount of chemicals required. Initially, 24 BV were used

²One Bed Volume (BV) is equal to the amount of resin in column, 1325 liters.

 ${\bf TABLE~2} \\ {\bf Cost~estimates~for~removal~of~Sr^{00}~for~various~BV~regenerants~per~440~liters~of~milk}$

Operating items	4.5	Cost for 3	BV used 14	24
	(\$)			
Raw materials	4.258	6.636	9.280	14.568
Labor	2.316	2.316	2.316	2.316
Utilities	.177	.177	.177	.177
Other operating expenses	.727	.727	.727	.727
Depreciation	.640	.640	.640	.640
Total	8.118	10.496	13.140	18.428

at 21 C. This figure was reduced to 14, then to 9 BV at 60 C, and finally to 4.5 BV at 60 C, according to the recommendation of Murthy (4). When using 4.5 BV of new regenerant, this solution is stored after passage through the column. It is then heated to 60 C and passed through the column on the succeeding run, to be discarded and then followed by 4.5 BV fresh regenerant. This method was used on the last run of the series; regeneration appeared to be adequate, although additional trials should be made to verify it.

Cost of the process was estimated per 440 liters of milk. These calculations consider only direct costs. Overhead costs, such as administrative, insurance, supervision, engineering, and plant floor space were not included. Labor costs include both operational and clean-up costs. These figures are based on 5,700 liters/

ur flow rates per 8-hr run and 260 runs per ar. Results are given in Table 2. These costs can be reduced by using a lower-priced grade of chemicals, by increasing the flow rate from 5,700 to 7,500 liters/hour, and by operating the plant on 90,000 liters instead of

45,400 liters of milk per day. All of these changes appear feasible.

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